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~~TITLE:~~ A Method, and Article of the Method, for Fabricating a Flexible, Hollow Waveguide.

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Field of the Invention ← Center

The present invention relates to waveguides and methods of making same, and more particularly to a flexible, rugged waveguide comprising a photonic, bandgap, hollow fiber, transmitting in the infrared and visible regions.

Initial Caps.

BACKGROUND OF THE INVENTION

The flexible waveguide of this invention is based upon technology disclosed in United States Patent Nos. 5,815,627; issued: September 29, 1998; 5,567,471, issued: October 22, 1996; and 5,440,664; issued: August 8, 1995, which technology and teachings are meant to be incorporated herein by way of reference. The aforementioned waveguide inventions have a common inventor, James Harrington, and a common assignee, Rutgers University. In aforementioned United States Patent No. 5,815,627, it was taught that waveguides are capable of guiding both IR and visible radiation. This is also a distinguishing feature of this invention. This invention,

however, has fabricated a waveguide that can be tailored for use over a wide wavelength range by the use of multiple, uniform, film coatings deposited by liquid phase chemistry methods. The coatings of the inventive waveguide have an additive, are fabricated by an additive technique rather than a subtractive method, i.e. the former Ag layer as taught in the aforementioned patents, was originally depleted in part, with the establishment of the AgI layer. The AgI layer is totally absent in the present application. Even the Ag layer is only present in one embodiment thereof, and is but one selection of other metals used for the metallic underlay.

This invention utilizes dielectric layers of sulfide materials, which are built in stacked geometries to fashion unique waveguide structures.

The waveguides of the present invention can be fabricated in one of two novel ways:

(a) The dielectric layers of cadmium and lead sulfide can be applied directly to the smooth bore surface of a silica glass tube; or

(b) The dielectric layers of the cadmium and lead sulfides can be applied to a metallic surface, for example, metals selected from a group consisting of Ag, Au, Cu, Pt, Ni, Pt, Mb, and Al, which has been smoothly coated upon the smooth inner bore of the silica glass tube.

A protective polymer coating is layered upon the outer surface of the tube in both embodiments. The dielectric layers of this invention comprise CdS and PbS, which form a unique compatibility pair, in which deposition of each material does not affect the underlying layer. These paired sulfides can be stacked in multiple, thin film layers to provide specific and unique characteristics. The sulfide layers are deposited using similar liquid-phase chemistry taught in the prior patents. The thickness of each layer can be tailored for use over a wide range of wavelengths. The measured losses for a single layer of CdS or a PbS film deposited over an Ag layer was in agreement with the prior Ag/AgI film studied at 10.6μm for applications using CO_2 IR lasers.

The CdS and PbS materials have disparate refractive indices with a ratio of about 2 to 1. This is often referred to in this technology, as the index contrast ratio. The high contrast of this layering makes possible a photonic bandgap hollow fiber. The deposit of the cadmium sulfide layer provides the transmission in the visible region.

Discussion of Related Art:

Prior-art hollow waveguides can provide poor beam quality (poor transverse spatial coherence). ~~FIG.~~ Figure 12B of Gregory & Harrington (see below cite) and ~~FIGS.~~ Figures 5 and 6 of Croitoru et al., "Characterization of hollow fibers for the transmission of infrared radiation", Appl. Opt. v. 29, 1805-1809 (20 Apr., 1990) and Dror et al., "Hollow Tubes for Transmitting IR Laser Energy for Surgery Applications", presented to ICALEO '89 (15-20 Jan., Los Angeles), are representative of the characteristics of prior art hollow waveguides.

United States Patent Nos. 5,815,627; issued: September 29, 1998; 5,567,471, issued: October 22, 1996; and 5,440,664; issued: August 8, 1995, depict Ag/AgI film waveguides. Typically, waveguides such as are disclosed in Matsuura & Miyagi, "Low-loss metallic hollow waveguides coated with

durable and nontoxic ZnS", Appl. Phys. Lett. v. 61, 1622-1623 (5 Oct., 1992) is superior. Waveguides with metal tube walls that serve as the supporting structure for any coatings (such as disclosed in U.S. Pat. No. 5,005,944, issued to Laakman et al., and U.S. Pat. No. 4,913,505, issued to Levy '505) may be capable of handling substantial power, but are semi flexible at best. Those with plastic tube walls (such as disclosed in U.S. Pat. No. 4,930,863, issued to Croitoru et al.) are flexible, but have marginal power-handling capability at best and high loss. Yet-earlier devices such as disclosed in U.S. Pat. No. 3,436,141, issued to Comte, U.S. Pat. No. 3,583,786, issued to Marcatili, and U.S. Pat. No. 3,963,828, issued to Onoda et al., have not proven useful for the applications of present interest at the wavelengths of present interest.

Hollow waveguide fibers having an index of refraction less than one, have not yet attained both transmission characteristics and flexibility required for many applications. However, in other respects these waveguide fibers are quite satisfactory at selected wavelengths. See Gregory & Harrington, "Attenuation, modal, and polarization properties of n<1, hollow dielectric waveguides", Appl. Opt. v. 32, 5302-5309 (20 Sept., 1993).

As aforementioned, the current invention is an improvement over the waveguide devices of the prior aforesaid Harrington patents.

The present invention has made loss measurements at key laser wavelengths, e.g. as a CO_2 laser waveguide. The current invention provides a flexible, hollow, waveguide, and method for making same. The waveguide tube meets the need for a flexible, visible and IR region, laser transmission medium having a relatively low loss.

SUMMARY OF THE INVENTION

Initial Caps

In accordance with the present invention, there is featured a flexible, hollow waveguide for the transmission of radiation in the infrared and visible regions, and a method of making same. The waveguide comprises a hollow, flexible, silica-glass tube having a transparent annular body defining a bore with a smooth inner bore surface. In

a first embodiment, a reflective, thin film, metallic layer, consisting of a metal selected from a group of metals consisting of: Ag, Au, Cu, Ni, Pt, Mb, Zn, and Al, is coated upon the smooth inner bore surface of the silica-glass tube. A single, thin film cadmium sulfide dielectric layer, or a pair of thin film dielectric, cadmium and lead sulfide layers respectively, are then disposed upon said reflective layer. Cadmium sulfide transmits radiation in both the visible and infrared region. This pair of sulfide layers has disparate refractive indices with a ratio of approximately 2 : 1, which is vital to provide high contrast, and to fabricate a photonic, bandgap, hollow waveguide tube. In a second embodiment, no metallic layer is used, and the pair of thin film cadmium and lead sulfide layers is multiply stacked directly upon the smooth, inner bore of the silica-glass tube.

The thin films are deposited using dynamic wet chemistry, and the thickness is tailored to minimize the attenuation of the waveguide over specific infrared wavelengths.

It is an object of the present invention to provide ~~a~~ an improved flexible, hollow waveguide.

It is another object of this invention to provide a flexible, hollow waveguide comprising a photonic, bandgap hollow fiber.

It is yet a further object of the current invention to provide a flexible, hollow waveguide, which transmits radiation in both the visible and infrared region.

BRIEF DESCRIPTION OF THE DRAWINGS

Initial Caps

A complete understanding of the present invention may be obtained by reference to the accompanying drawings, when considered in conjunction with the subsequent detailed description, in which:

FIGURE 1 depicts the growth kinetic graph of the thin films deposited in the Ag coated 1,000-μm bore waveguide;

FIGURE 2 shows the graph of the UV-VIS spectra of a 1,000-μm bore Ag/CdS waveguide

FIGURE 3 illustrates the graph of the FTIR spectra of a 1,000-μm bore Ag/Pbs waveguide;

FIGURE 4 depicts a graph of the cross-sectional FESEM image of a 1,000-μm bore Ag/CdS/PbS waveguide;

FIGURE 5 shows a graph of the FTIR spectra of a 1,000-μm bore of Ag/CdS and Ag/PbS, Ag/CdS/PbS and Ag/CdS/PbS/CdS waveguide;

FIGURE 6 illustrates a partial schematic view of the first embodiment of the waveguide of this invention;

FIGURE 6a depicts a partial schematic view of the second embodiment of the waveguide of this invention;

DESCRIPTION OF THE PREFERRED EMBODIMENT

Generally speaking, a flexible, hollow, waveguide is featured, that functions in the infrared and visible regions. The waveguide comprises a hollow, flexible, silica-glass tube having a smooth borethat is coated in one embodiment with a reflective, metal substance on the inner bore surface. A pair of sulfide materials respectively of cadmium and lead sulfide, is then layered over the reflective substance. The sulfide materials form a high contrasting refractive index of approximately 2 : 1, thus creating a photonic, bandgap tube. In a second embodiment, the metallic layer is not used, and the pair of sulfide layers is singularly or multiply stacked directly upon the bore.

There are two embodiments of the invention. The first embodiment fabricates the waveguide by liquid phase deposition of a metallic, reflective layer, usually Ag upon the smooth inner bore surface of the silica-glass tube. Other metals of choice such as Au, Cu, Ni, Pt, Zn, Mb, Al, etc., can also be used. Then, a single or pair of sulfide-containing dielectrics is coated over the metal. In the second embodiment of the invention, no metallic or reflective layer

is deposited, and only pairs of sulfide-containing dielectric films are coated upon the smooth inner bore.

Now referring to ~~FIGURE~~ Figure 6, a first embodiment of the waveguide 1 of this invention is illustrated. The waveguide 1, comprises a silica-glass tube 3, having a thin-wall of approximately 50 to 200 microns wall thickness, and a smooth inner bore. A reflective, metallic layer 4 is coated over the smooth bore using liquid phase chemistry, as taught in the aforementioned patents. The reflective layer may comprise Ag, Au, Ni, Cu, Al, Pt, Zn, Mb, etc. A single layer of cadmium sulfide, or paired sulfide composite of cadmium sulfide and lead sulfide, respectively, is layered over the reflective layer 4. The sulfide composite comprises respective layers 5 and 6, of cadmium and lead sulfides, respectively. The sulfide materials form a high contrast, refractive index of approximately 2 : 1 creating a photonic, bandgap tube. The silica-glass tube 3 is covered with an outer layer 2 of plastic for protection.

Referring to FIGURE 6a, a waveguide ~~10~~ 1a is shown having a flexible, hollow, silica-glass tube ~~11~~ 3, upon which is stacked at least one pair of cadmium and lead sulfide layers ~~12~~ 5, and ~~14~~ 6, respectively. A multiplicity of pairs of cadmium and lead

sulfide layers 5 and 6 can be deposited over the first pair, as shown in phantom.

Examples: NO Bold

1. A smooth, inner bore surface 3 of a flexible hollow tube 2 of a waveguide 1, is coated with a metallic, reflective layer 4 of silver. The silver layer 4 was then coated with cadmium sulfide to form layer 5, and then coated with lead sulfide to form layer 6. Each layer was coated using liquid phase chemistry, utilizing a peristaltic pump. The flow rate of the solutions through the hollow tube was chosen at 30 ml/min.

The cadmium sulfide coating was applied over the Ag coating, using one of the two chemical baths.

1. I) Cadmium Nitrate 0.1 M, II) thiourea 0.5 M - this is considered a full concentration solution. For most depositions, the concentration was reduced to 1/2 to 1/6 the full concentration.

2. I) Cadmium acetate 5mM, II) thiourea 100mM.

All the coating solutions were prepared in distilled and deionized water. The solutions are prepared using an ultrasonicator or a magnetic stirrer.

The Cd ion containing solution is complexed with ammonium hydroxide solution. Initial addition of ammonia will form a white precipitate of $Cd(OH)_2$ and the solution becomes turbid. With further addition of ammonia dissolves the white precipitate and forms a soluble Cd-ammonia complex. The solution pH is maintained between 10 and 13 using sodium hydroxide solution and nitric acid as titrating agents. The second solution of sulfide ion is prepared by dissolving thiourea in water. The two solutions were then flowed through the hollow tube and coated the Ag. The complexed Cd ion solution reacts with the sulfide ion containing solution to deposit a thin film of CdS. The hollow tube was dried in flowing air.

2. For deposition of PbS thin films, lead nitrate was used as the source of Pb ions and thiourea or a mix of

thiourea and thioacetamide was the source of sulfide ions. All the solutions were prepared in distilled and deionized water (DI). The solutions are prepared using an ultrasonicator or a magnetic stirrer. The chemical bath used for the deposition of lead sulfide is as given below:

Solution 1: A) $Pb(NO_3)_2$: 4g / 500 ml; b) NaOH: 12 g/500ml) and

Solution 2: thiourea: 6g/ 1000 ml or thiourea 4g and thioacetamide 2 g to 1000ml of water.

The NaOH solution is slowly added to the $Pb(NO_3)_2$ solution while continuing to stir. Initially the solution turns turbid due to the precipitation of $Pb(OH)_2$ which dissolves to form a soluble Na-Pb-hydroxide complex on further addition of NaOH. The complexed lead ion reacts with a sulfide ion in solution to form PbS. Deposition of PbS requires that the solutions containing Pb and S ions are supersaturated in order to precipitate out in solution and nucleate heterogeneously on the substrate. The bore surface of the waveguide was coated with the solutions using the peristaltic pump as previously shown. Surfaces were air dried.

3. Infrared and visible region investigation of Ag/CdS, Ag/PbS, Ag/CdS/PbS and Ag/CdS/PbS/CdS waveguides, as can be observed with reference to the FIGURES 1 through 3 and 5X, will now be described.

In the original work on omni directional waveguides authored by Fink, et al.[3] (insert reference "3" info. from p. 26) an all-dielectric structure of alternating low/high index films was described. In this invention, use of a metallic film in conjunction with a multilayer dielectric stack means that the waveguide will need fewer dielectric layers to achieve the same loss as an all dielectric omni directional structure. CdS and PbS films are transparent in the 2 to 12 μm region. The refractive indices of CdS and PbS are 2.25 and 4.27 at 1.55 μm and 2.25 and 4.0 at 10.6 μm, respectively. This gives an index contrast of 4.27/2.25 = 1.9 at 1.55 μm and 1.78 at 10.6 μm. These two dielectric materials and other sulfides such as ZnS and ZnSe may be deposited in thin film form using straightforward solution chemistry methods.[4-6] (insert refs. 4+6 from p. 26) Furthermore, these two materials are compatible, and the wet chemistry methods used to deposit both films are similar.[7,8] (insert refs. 7+8 from p. 26)

Dielectric-coated metallic hollow waveguides of this invention are designed to minimize the attenuation of the waveguide over a particular IR wavelength region by optimizing the thickness of each dielectric layer. The well-established theory of Miyagi and Kawakami (insert ref. 9 from page 27) was used to calculate the expected losses for multilayers of CdS and PbS coatings. Using this theory, and the n and k values for CdS and PbS, the losses for single and multilayer films at both 1.55 and 10.6 μm were calculated. The calculations at 10.6 μm show that a 1,000-μm bore of a hollow glass waveguide with a 3-layer stack ~~stac~~ of CdS/PbS/CdS films deposited over Ag will have a straight loss of 0.016 dB/m compared to a straight loss of 0.07 dB/m for a single layer CdS film deposited over Ag. That is, the calculated attenuation coefficients are approximately four times less for the three layer design, compared to a single dielectric layer at 10.6 μm.

As aforementioned, hollow glass waveguides are prepared in a two step process in which an Ag film is first deposited on the inner surface of the silica tubing and then a dielectric layer of CdS or PbS is deposited on top of the metallic layer. For multilayer dielectric structures, sequential deposition of alternating low/high films leads to the structures, Ag/CdS, Ag/CdS/PbS, and Ag/CdS/PbS/CdS. The Ag film is deposited on the inner surface of the silica tubing using a liquid-phase reduction

reaction typical of that used in the prior art patents to Harrington (insert refs. 10 & 12 from A27) using Ag/AgI hollow glass waveguides [10-12]. The thickness of the Ag film is chosen to be sufficiently thick to prevent any transmission through the film, but thin enough so that the surface roughness is as low as possible. Specifically, the thickness of the Ag film is chosen to be at least ten times the skin depth at 10.6 μm. The skin depth of an Ag film at 10.6 μm is 12 nm. In all the experiments CdS and PbS have been deposited on Ag films that vary in thickness from 150 to 200 nm. At this thickness the Ag films are quite smooth (< 12 nm rms roughness) and, therefore, they provide a good surface for the deposition of the dielectric layers. A very important feature of the CdS and PbS coatings is that they are additive and, thus, independent of the Ag film. This is in strong contrast to the waveguides made using AgI coatings (insert refs. 11, 13, and 14 from page 27 to 28). AgI is deposited in a subtractive process in which AgI is formed by the diffusion of iodine ions into the underlying silver film. Therefore, to produce a thick dielectric layer of AgI will require a thick starting layer of Ag. In an article by Rabii and Harrington [15] they have shown that the surface roughness of both the Ag and the AgI film increases with increasing thickness of the Ag layer.

Additional Examples: Move to next page, No Bold

As aforementioned, CdS and PbS thin films were deposited using a wet chemistry deposition technique (Suggest 4 lines p.26). Cadmium nitrate and cadmium acetate are used as the source of Cd ions; lead nitrate the source of Pb ions; and thiourea, $(SC(NH_2)_2)$, the source of S ions. It is important to accurately control pH of these solutions, since the CdS and PbS precipitates are stable only in the pH range 10 to 13. The pH control and chelating is achieved using an ammonia solution for CdS and sodium hydroxide for PbS. Specifically, the pH for aqueous $Cd(NO_3)_2$ is maintained between 11 and 12. Owing to the fact that the reaction rate also changes with the pH, it is necessary to calibrate the process for a given pH range. The thickness of both CdS and PbS increases with increasing concentration of Cd, Pb and S ions in solution and with increasing deposition time. From a study of the growth kinetics for both CdS and PbS thin films, it has been established, that the optimal concentration and flow rate conditions for uniform film deposition for tubing require lengths greater than 1.5 m. The best coatings were made using > 0.01 M solutions and pumping rates of 30 ml/min.

The multilayer dielectric structures of Ag/CdS/PbS and Ag/CdS/PbS/CdS, were prepared in a manner similar to the single-

layer dielectric metallic waveguides. The different layers were coated in a sequential manner with an intermediate drying step after coating each layer. An Ag-only tube was coated with a single layer of either CdS, or PbS, when the 2- and 3-layer structures were being coated as an independent check on the thickness for each layer.

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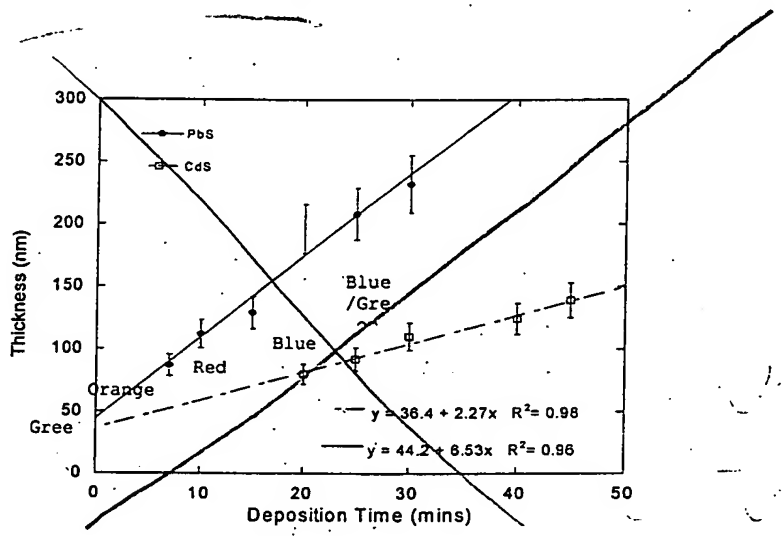
Optical characterization of Ag/sulfide film Waveguides:

The spectral characteristics of the Ag/CdS and Ag/PbS hollow glass waveguides were evaluated using a Perkin Elmer UV-VIS spectrometer, and Nicolet Protégé FTIR. A typical spectral response shows interference peaks, which depend on the thickness of the dielectric thin films. The thickness of a single layer dielectric, "d", was calculated from the peak position of the longest-wavelength interference band, $\lambda_p^{(m)}$, using the relation [32],

$$d = \frac{m \cdot \lambda_p^{(m)}}{4 \cdot \sqrt{n_1^2 - 1}}, \qquad (1)$$

where m is the order of the interference maxima ($m=1$ for the longest-wavelength band); λ_p is the wavelength of the m^{th} absorption peak; and n_1 is the refractive index of the dielectric film. From Equation (1) it is observed, that the peak position shifts to longer wavelengths as the thickness of the film

increases. Spectral data has been used to determine the thickness of films prepared using different growth kinetics. The film thickness obtained from the optical measurements has then been correlated with direct thickness measurements using a field-emission scanning electron microscope (FESEM). In this way one obtains the growth kinetic curves for CdS and PbS deposited on Ag, as shown in ~~Fig. 1.~~ Figure 1.



In Figure 1, the ~~Fig. 1 The~~ growth kinetic curves of the CdS and PbS thin films deposited in a Ag coated, 1,000 μm bore Hollow Glass Waveguide, are show.

datum points of curves 60 and 62 represent

The ~~color indicated is the~~ color regions that one would see looking through the waveguides with an optical ~~microscope.~~ microscope, and are representative of film thickness.

All films were prepared using 0.01 M solutions and a flow rate of 30 ml/min. The growth kinetics curves in ~~Fig.~~ Figure 1 indicate that

CdS (open symbols) has a slower growth rate on Ag compared to PbS (solid symbols) under similar conditions. The reason for the slower growth rate for CdS compared to PbS, is that the CdS complexes with the ammonia used in the deposition and this decreases the rate of deposition. The data also shows that the thickness of both dielectrics increases linearly with time, with growth rates of ~ 2.3 and 6.9 nm/min for CdS and PbS, respectively. The mechanism for linear growth is based on the Stransi-Krastanov model of island-like growth (Insert from p. 28) [19]. This mechanism involves nucleation and growth in the linear growth region. This information is important as we need to carefully control film thickness for single and multilayer structures. The UV-VIS spectra for the Ag/CdS is given in ~~Fig. 2,~~ Figure 2, and the FTIR spectra for the Ag/PbS in ~~Fig.~~ Figure 3. The spectral data clearly show that the position of the interference peaks (50, 51, 52, and 53, respectively) shifts to longer wavelengths as the thickness of the film increases as predicted by ~~Eq.~~ Equation (1). It ~~is~~ has also ~~noted from the insert photos in Fig. 2,~~ been observed relative to Figure 2, that the Hollow Glass Waveguides show a color variation. This is due to selective filtering of the input white light by the thin film coating (interference effect). This color variation is seen with the CdS coatings, but not the PbS films, because these films do not transmit well at visible wavelengths.

The thickness of the thin films was obtained by direct measurements using a FESEM. A photomicrograph taken with the FESEM for a typical cross-section of the thin film combination on a silica substrate 44 of Ag/CdS/PbS identified as 45, 46, and 47, respectively, is shown in Figure 4. The CdS film appears darker than the PbS film, because the in-lens detector produces a negative image of the secondary electron image. Also, the silica substrate 44 is a portion of the tube 3. The thicknesses of the films shown in Figure 4 are; Ag 154 nm, CdS 169 ± 16 nm, PbS 82 ± 6 nm. Degradation was not observed for the underlying film, when the new film is deposited over it. That is, CdS and PbS do not react with each other during the deposition of successive layers. The film thickness measured from the FESEM micrographs of both single and multilayer dielectric film are summarized in Table 1, below. These results agree very well with the optical thickness measurement.

Table 1. Thickness values for CdS and PbS thin films on Ag determined from FESEM images

HGWs	Thickness of Ag nm	Thickness of CdS nm	Thickness of PbS nm
Ag/CdS	156	172 ± 16	—
Ag/PbS	158	—	96 ± 16
Ag/CdS/P	154	169	82 ± 6

A series of 1,000-μm-bore HGWs with 1, 2, and 3 dielectric layers deposited over Ag were fabricated using wet chemistry methods [8, 11]. The spectral losses for these straight waveguides are shown in ~~Fig.~~ Figure 5. From ~~Fig.~~ Figure 5, it may be seen that the addition of each dielectric layer shifts the interference peaks 56, 57, 58, respectively, to longer wavelengths. This is a result of the increase in thickness with each additional layer.

The thickness of each dielectric layer was determined from a ~~wi~~ witness sample composed of each dielectric layer deposited separately on a slide. These witness samples were deposited along with the multilayer ~~waveg~~ waveguides. From the witness samples we determined the thickness of the ~~indiv~~ individual layers using the position of the long wavelength interference peak from Equation ~~Eq.~~ (1). The thicknesses obtained were; Ag 200 nm, CdS (adjacent to Ag) 156 nm, PbS 87 nm, and CdS (next to air) 97 nm. The thickness values of CdS and PbS layers measured optically agree very well with FESEM measurements and are within experimental errors as shown in Table 1. From ~~Fig.~~ Figure 5 we see that this waveguide is best suited for operation beyond 5 μm.

Losses measurements were made at 1.55 μm using a diode laser rather than a CO_2 laser at 10.6 μm where the HGWs would ultimately be most useful. The reason for using a 1.55 μm laser was that we were ~~interested~~ interested to

developing a new waveguide for secure communication systems at 1.55

Clearly, solid-core silica fibers are a better choice for applications at 1.55 μm. The output of the diode laser was a pigtailed single mode fiber terminated with a Selfoc lens. The response of the HGWs chosen for loss measurements was similar to shown in Fig. 5. In general, coating thicknesses were not optimized for lowest loss at 1.55 μm; however, as may be seen from the cut-back data given in Table 2, the losses were still quite low. The losses in Table 2 may be compared to the theoretical losses calculated both 1.55 and 10.6 μm using the n and k values of the dielectric and the theory of Miyagi and Kawakami [9]. The losses at 1.55 μm are high when PbS is used because k is rather large at this wavelength (k=0). At 10.6 μm k for PbS is 0.008 and the calculated loss for Ag/CdS/PbS is over four times less than for Ag/CdS. The bending losses for waveguides were not measured. It is well known that there is an additional loss on bending for non-omni directional waveguides, which varies as 1/R, where R is the radius of the bend [12]. Since our waveguides have no more that 3 dielectric layers it is expected that we would not observe omni directional behavior rather there would be added loss on bending.

Table 2 Loss values for 1,000-μm bore HGWs with 1, 2, and 3-layer dielectric coatings.

Multilayer	Measured l· μm,	Theoretic 1.55 μ	Theoretic 10.6 μ
Ag/CdS	0.2 ±	2.7	7.0
Ag/PbS	0.26 :	2.5	7.3
Ag/CdS/PbS	0.1 ±	8.6	3.3
Ag/CdS/PbS/Cc -	0.06 :	6.7	1.6

demonstrated that liquid-phase chemistry methods can be used to deposit good optical quality CdS and PbS thin films to form both single and multiple dielectric/metallic HGWs. The spectral response for waveguides with these films deposited over Ag show well defined interference bands indicating good film thickness uniformity over the entire length of the guide. Varying the deposition time controls the position of the interference peaks. Furthermore, we have found that CdS and PbS are compatible and the deposition of each material does not affect the underlying film. In this way the thickness of each layer in the multilayer stack can be tailored for use over a wide wavelength range. The final 3-layer stack showed that it is possible to make a multilayer coating but we have yet to reduce the attenuation to the level predicted by theory. Moreover, the measured losses for the single-layer CdS or PbS films at 1.55 μm are in general agreement with the well studied Ag/AgI HGWs at 10.6 μm [17]. In reality, the greatest potential for these waveguides is likely to

be at 10.6 μm for applications involving CO_2 laser power delivery and IR fiber sensors.

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Since other modifications and changes varied to fit particular operating requirements and environments will be apparent to those skilled in the art, the invention is not considered limited to the example chosen for purposes of disclosure and covers all changes and modifications which do not constitute departures from the true spirit and scope of this invention.

Having thus described the invention, what is desired to be protected by Letters Patent is presented in the subsequently appended claims.

~~What is claimed is:~~

DRAWINGS

CdS and PbS thin films were deposited using a wet chemistry deposition technique [4]. Cadmium nitrate and cadmium acetate are used as the source of Cd ions; lead nitrate the source of Pb ions; and thiourea, $(SC(NH_2)_2)$, the source of S ions. It is important to accurately control pH of these solutions since the CdS and PbS precipitates are stable only in the pH range 10 to 13. pH control and chelating is achieved using an ammonia solution for CdS and sodium hydroxide for PbS. Specifically, the pH for aqueous $Cd(NO_3)_2$ is maintained between 11 and 12. Since the reaction rate also changes with the pH, it is necessary to calibrate the process for a given pH range. The thickness of both CdS and PbS increases with increasing concentration of Cd, Pb and S ions in solution and with increasing deposition time. From a study of the growth kinetics for both CdS and PbS thin films, we have established optimal concentration and flow rate conditions for uniform film deposition for tubing with lengths greater than 1.5 m. The best coatings were made using > 0.01 M solutions and pumping rates of 30 ml/min.

The multilayer dielectric structures of Ag/CdS/PbS and Ag/CdS/PbS/CdS, were prepared in a manner similar to the single-layer dielectric metallic waveguides. The different layers were coated in a sequential manner with an intermediate drying step after coating each layer. An Ag-only tube was coated with a single layer of either CdS or PbS when the 2- and 3-layer structures were being coated as an independent check on the thickness for each layer.

3. Optical characterization of Ag/sulfide film HGWs

The spectral characteristics of the Ag/CdS and Ag/PbS HGWs were evaluated using a Perkin Elmer UV-VIS spectrometer and Nicolet Protégé FTIR. A typical spectral response shows interference peaks which depend on the thickness of the dielectric thin films. The thickness of a single layer dielectric, d, was calculated from the peak position of the longest-wavelength interference band, $\lambda_p^{(m)}$, using the relation [12],

$$d = \frac{m \cdot \lambda_p^{(m)}}{4 \cdot \sqrt{n_1^2 - 1}}, \qquad (1)$$

where m is the order of the interference maxima ($m = 1$ for the longest-wavelength band); λ_p is the wavelength of the m^{th} absorption peak; and n_1 is the refractive index of the dielectric film. From Eq. (1) we see that the peak position shifts to longer wavelengths as the thickness of the film increases. Spectral data has been used to determine the thickness of films prepared using different growth kinetics. The film thickness obtained from the optical measurements has then been correlated with direct thickness measurements using a field-emission scanning electron microscope (FESEM). In this way we obtain the growth kinetic curves for CdS and PbS deposited on Ag, shown in Fig. 1.

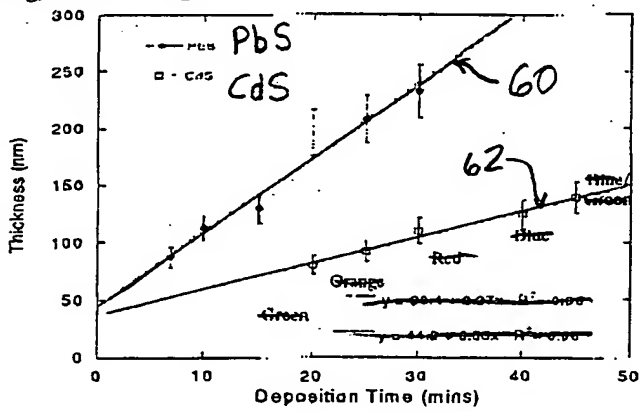


Fig. 1 The growth kinetic curves of the CdS and PbS thin films deposited in a Ag coated, 1,000 μm bore HGWs. The color indicated is the color that one would see looking through the waveguides with an optical microscope.

FIG. 1

All films were prepared using 0.01 M solutions and a flow rate of 30 ml/min. The growth kinetics curves in Fig. 1 indicate that CdS (open symbols) has a slower growth rate on Ag compared to PbS (solid symbols) under similar conditions. The reason for the slower growth rate for CdS compared to PbS is that the CdS complexes with the ammonia used in the deposition and this decreases the rate of deposition. The data also show that the thickness of both dielectrics increases linearly with time, with growth rates of ~ 2.3 and 6.9 nm/min for CdS and PbS, respectively. The mechanism for linear growth is based on the Stransi-Krastanov model of island-like growth [16]. This mechanism involves nucleation and growth and we are in the linear growth region. This information is important as we need to carefully control film thickness for single and multilayer structures. The UV-VIS spectra for the Ag/CdS is given in Fig. 2 and the FTIR spectra for the Ag/PbS in Fig. 3. The spectral data clearly show that the position of the interference peaks shifts to longer wavelengths as the thickness of the film increases as predicted by Eq. (1). We also note from the insert photos in Fig. 2 that the HGWs show a color variation. This is due to selective filtering of the input white light by the thin film coating (interference effect). This color variation is seen with the CdS coatings but not the PbS films as these films do not transmit well at visible wavelengths.

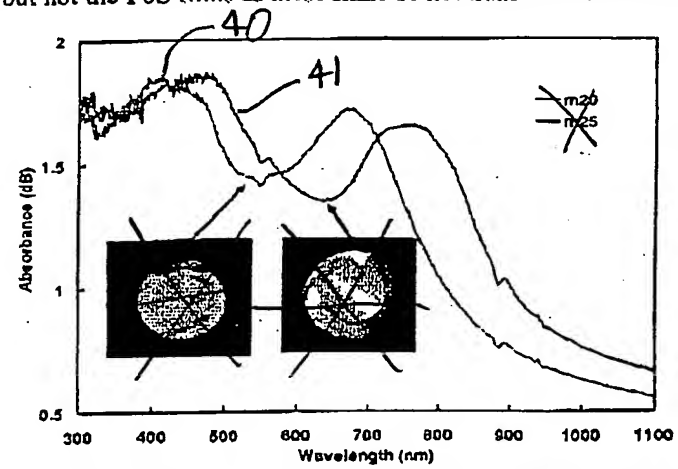


Fig. 2 UV-VIS spectra of 1,000-μm-bore Ag/CdS HGWs for different deposition times in m minutes. The insert shows the color of the transmitted light as filtered by the coating.

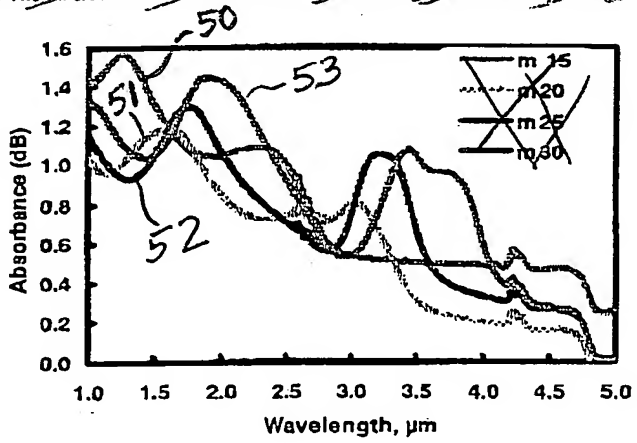


Fig. 3 FTIR spectra of 1,000-μm bore Ag/PbS HGWs for different deposition-times in m minutes.

The thickness of the thin films was obtained by direct measurements using a FESEM. A photomicrograph taken with the FESEM for a typical cross-section of the thin film combination Ag/CdS/PbS is shown in Fig. 4. The CdS film appears darker than the PbS film because the in-lens detector produces a negative image of the secondary electron image. The thicknesses of the films shown in Fig. 4 are; Ag 154 nm, CdS 169 ± 16 nm, PbS 82 ± 6 nm. We do not see any degradation of the underlying film when the new film is deposited over it. That is, CdS and PbS do not react with each other during the deposition of successive layers. The film thickness measured from the FESEM micrographs of both single and multilayer dielectric film are summarized in Table 1. These results agree very well with the optical thickness measurement.

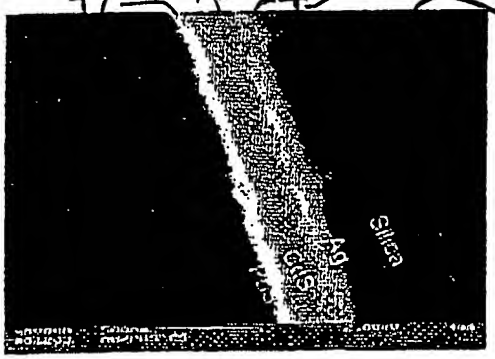


Fig. 4 Cross-sectional FESEM image of a 1,000-μm bore Ag/CdS/PbS HGW. The CdS and PbS thin films are deposited sequentially inside the Ag coated HGW.

Table 1 Thickness values for CdS and PbS thin films on Ag determined from FESEM images

HGWs	Thickness of Ag nm	Thickness of CdS nm	Thickness of PbS nm
Ag/CdS	156 ± 16	172 ± 16	—
Ag/PbS	158 ± 20	—	96 ± 16
Ag/CdS/PbS	154	169	82 ± 6

A series of 1,000-μm-bore HGWs with 1, 2, and 3 dielectric layers deposited over Ag were fabricated using wet chemistry methods [8, 11]. The spectral losses for these straight waveguides are shown in Fig. 5. From Fig. 5, it may be seen that the addition of each dielectric layer shifts the interference peaks to longer wavelengths. This is a result of the increase in thickness with each additional layer.

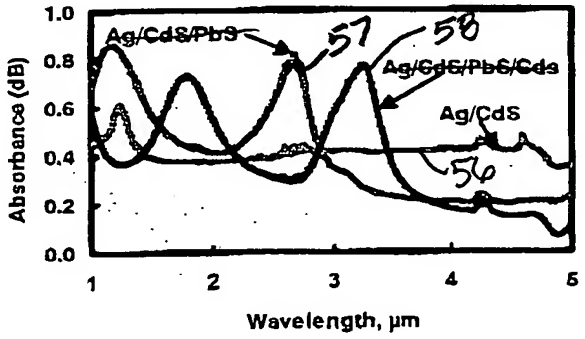
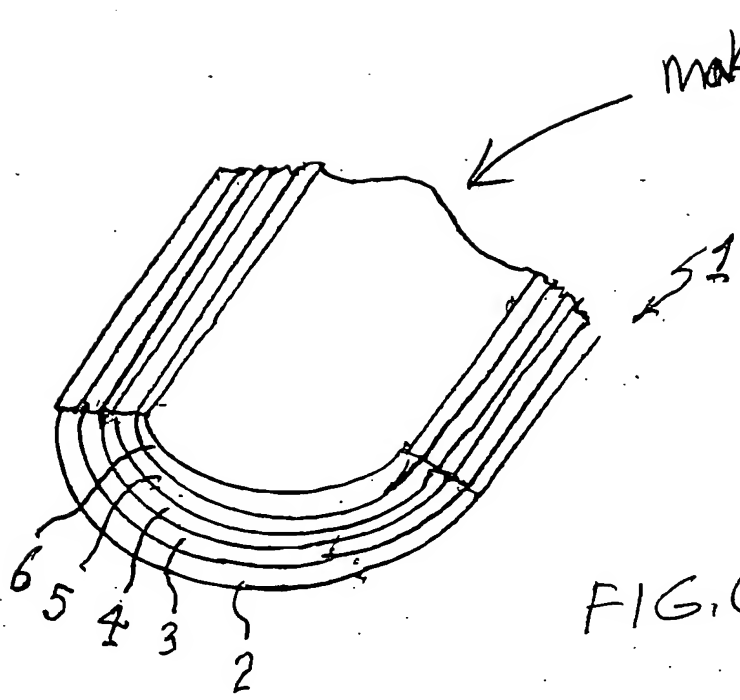


Fig. 5 FTIR Spectra of 3 1,000-μm bore of 1, 2, and 3-layer dielectric coatings on Ag coated HGWs. The spectra beyond 4 to 12 μm region are essentially flat and featureless.



~~FIGURE 6~~

FIG. 6

~~1a~~

FIGURE 6a

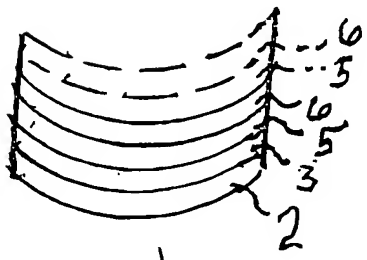


FIG. 6a

make Rounder